

PHILIP MORRIS INC.
RESEARCH AND DEVELOPMENT DEPARTMENT

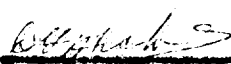
Completion Report 19

Project 35-1301

IMPROVEMENT IN BL AS MANUFACTURED

Runs 163, 164, 167-69, 169-71

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Richmond, Virginia

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I. Summary

The overall objective of this project is to improve the physical characteristics and the smoke flavor of BL. The ultimate would be a blended leaf indistinguishable from natural leaf tastewise, and superior to natural leaf physically.

The immediate objectives of these experiments are to determine the effect of the substitution of triethylene glycol and propylene glycol for glycerine in the binder on:

- (a) physical characteristics
- (b) smoke flavor
- (c) coal strength
- (d) TPM and nicotine delivery
- (e) mold inhibition

The objectives also include the effect of the use of propylene glycol as an overspray on the flavor. Preliminary smoke tests indicated that the use of 6% propylene glycol on BL resulted in a smoother, milder smoke.

The change in binder formulation did not affect the physical properties of the BL, the smoke flavor, or the TPM and nicotine delivery. The coal strength of cigarettes made from BL 164 (propylene glycol binder) was greater than the coal strength of cigarettes made from BL with triethylene glycol binder, glycerine binder, or propylene glycol binder with propylene glycol overspray.

The use of propylene glycol as an overspray (169-71) reduced the tensile strength of the BL, improved the smoke flavor of BL cigarettes, increased slightly the TPM delivery of BL cigarettes, and reduced the coal strength. The reduced tensile strength and coal strength may be a result of the product being over plasticized. The target weight of propylene glycol in run 169-71 was 8-9%. The actual weight was 11%. The overspray experiment is to be re-run at a lower level of propylene glycol.

The bacteriological study showed that the use of propylene glycol in the binder provides greater mold inhibition than does triethylene glycol or glycerine, and the use of propylene glycol as an overspray provides greater protection than does the propylene glycol in the binder.

II. Conclusions

Based on the evaluations of these samples, the following conclusions can be drawn:

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- (a) The substitution of propylene glycol for glycerine in the binder improves the mold inhibition and coal strength, and does not change the physical properties, smoke flavor, or TPM and nicotine delivery.
- (b) The use of propylene glycol (5% level) overspray on BL which contains propylene glycol in the binder improves smoke flavor, reduces tensile strength, increases TPM delivery, and improves mold inhibition.

Based on these conclusions, it is recommended that the overspray experiment (169-71) be re-run at propylene glycol target weight of 8% for complete reevaluation of the BL.

III. Procedures, Results, and Discussions

The pilot plant operation followed established procedures. The procedures and operating data are recorded in Engineering Notebook 114 pages 61 to 56, 72, 74, and 75, and in R & D notebook 35-1301, book V, pages 29, 30, 36, and 37, copies of which are appended to the original copy of this report. A summary of the materials used in these experiments is appended as exhibit A.

Standard procedures of the Development Division were used in the physical evaluations of the samples, and the results are appended as exhibit B. The physical characteristics are within the established limits except for the tensile strength of BL sample 169-71. The low tensile could be a result of the high plasticizer content of the sample. The target weight of propylene glycol in sample 169-71 was 6%. Preliminary experiments indicated that we might expect as much as 50% loss of propylene glycol upon redrying the sample, the actual loss of propylene glycol through "C" stage dryer was 41%.

Propylene glycol was used to inhibit mold growth, and propylene glycol was an overspray to improve the flavor. Bacteriological studies show that sample 164 (propylene glycol binder) is superior to 163 (triethylene glycol binder) and 167-69 (glycerine binder) in mold resistance, and sample 169-71 (propylene glycol binder and overspray) is a superior mold inhibitor than the other three samples (see exhibit C).

Smoking tests show that cigarettes made from sample 169-71 are milder and are preferable to cigarettes made from 163, 164, and 167-69 (see exhibit D).

The rapid smoke method shows that sample 169-71 is slightly higher in TPM delivery than 167-69, but there is no difference in nicotine delivery (see exhibit E). The slight increase in total particulate matter delivered by sample 169-71 may be a result of the high plasticizer level.

* This conclusion is based on the limited data presented in Exhibit F.

Cigarettes made from experimental EL 164 proved to have coal strength superior to cigarettes made from the EL of runs 163, 167-69, and 169-71. The results are shown in exhibit F.

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PILOT PLANT BL MATERIAL SUMMARY

- A. 163 Triethylene Glycol Binder
- B. 164 Propylene Glycol Binder
- C. 167-19 Glycerine (STD) Binder
- D. 169-21 STD Binder - Propylene Glycol Over spray

| DUST | A | B | C | D |
|------------|------|------|------|------|
| TYPE | PROD | PROD | PROD | PROD |
| FLAVORS | 4% | 4% | 4% | 4% |
| GRIND | -50M | -50M | -50M | -50M |
| SIZE DIST. | | | | |

BELT WATER

| | | | | |
|--------------|-----|-----|-----|-------------------------|
| ORVUS % | — | — | — | — |
| ALL % | 0.1 | 0.1 | 0.1 | 0.1 |
| HONEY % | — | — | — | — |
| CORN SYRUP % | 3 | 3 | 3 | 3 |
| GUM CONTENT | — | — | — | — |
| PH | — | — | — | — |
| Other | | | | *10.5% Propylene Glycol |

TRUCK

| | | | | |
|-----------------------|------|------|-------------|------|
| MADE | PP | PP | PROD | PP |
| PH | 2.75 | 2.6 | — | — |
| SOLIDS, % | 3.07 | 3.03 | — | — |
| VISCOSITY, CPS | 5000 | 4500 | — | — |
| TYPE | PROD | PROD | PROD | PROD |
| PULP TO GUM | 1-1 | 1-1 | 1-1 | 1-1 |
| GMS / FT ² | 100 | 110 | 10 (Approx) | 100 |
| Residual | TEG | PG | Est. | PG |

* In Humidification Water Only

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PILOT PLANT BL EVALUATION FLAVOR IMPROVEMENT PROGRAM

A-1163 TRIETHYLENE GLYCOL BINDER

B-124 PROPYLENE GLYCOL BINDER

C-16769 GLYCERINE (STD) BINDER

D-16771 STD(PG) BINDER # PROPYLENE GLYCOL OVERSPRAY

| TEST | PRODUCTION STANDARDS | A TEST | STD DEV. | B TEST | STD DEV. | C TEST | STD DEV. | D TEST | STD DEV. |
|--|-------------------------|-----------|-------------|-----------|-------------|-----------|-------------|------------|-------------|
| BASIS WEIGHT, GMS/FT ² | 10-11 | 9.7 | 0.15 | 9.1 | 0.25 | 9.5 | 0.21 | 8.7 | 0.22 |
| MOISTURE, % | 12-13 | 12.6 | 0.48 | 12.9 | 0.28 | 12.7 | 0.23 | 13.0 | 0.11 |
| TENSILE STRENGTH, KG/IN | 0.6 MIN | 0.80 | 0.06 | 0.76 | 0.06 | 0.76 | 0.04 | 0.43* 0.07 | |
| TEAR STRENGTH, GMS | 4.0 MIN | 4.9 | 0.53 | 4.1 | 0.55 | 4.9 | 0.81 | 4.3 | 0.72 |
| DRST LOSS (CUTTING), GMS/FT ² | 2.0 MAX. | 0.91 | 0.05 | 0.91 | 0.05 | 1.0 | 0.07 | 0.82 | 0.02 |
| DRST OFF, GMS/FT ² | 1.5 MAX. | 0.53 | 0.06 | 0.52 | 0.03 | 0.54 | 0.05 | 0.66 | 0.04 |
| BREAKAGE, % - 20M | 30 MAX. | 21.4 | 2.8 | 28.6 | 3.2 | 23.8 | 5.3 | 18.4 | 6.1 |
| FILLING POWER, CC/10GM | 35 MIN | 38.0 | 0.0 | 38.0 | 0.0 | 38.0 | 0.0 | 34 | 0.0 |
| BURNING RATE, MG/SEC | 2.0 MAX. | 1.8 | 0.07 | 1.7 | 0.05 | 1.6 | 0.5 | 1.6 | 0.19 |
| BURNING RATE, % BURNED | 95 MIN | 96.4 | 0.47 | 96.1 | 0.31 | 95.9 | 0.55 | 94.7 | 1.5 |

TOTAL PLASTICIZER %

TEG
None DetectedPG
2.8GLYCERIN
2.7PG
11

RUN OF TENSILE STRENGTH - 0.43 KG/IN

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Dr. C. B. Saligman

August 20, 1959

J. C. Malone

Study of Mold Inhibition by Propylene Glycol.

Attached is Mrs. Johnson's report of our study of the mold inhibitory properties of propylene glycol in BL. The results of these tests indicate that the application of propylene glycol both in the binder and the over spray is the most effective treatment. Propylene glycol in the binder only is more effective than either glycerine or triethylene glycol.

If you desire further information, please contact me.

JCH:mar

cc: Dr. L. S. Harrow
Mr. L. L. Long
Mrs. Virginia Johnson

J. C. Malone

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SPOT TEST OF TREATED BL.

| <u>MUSCANTANTS</u> <u>NO. OF DAYS</u> | <u>GLYCERINE</u> | <u>TEO</u> | <u>PROPYLENE GLYCOL</u> <u>IN BINDER</u> | <u>PROPYLENE GLYCOL IN</u> <u>BINDER AND OVERSPRAY</u> | <u>CONTROL AGAR</u> |
|--|--|------------------------|---|---|---------------------------------------|
| 1 | Beginning to mold | No mold | No mold | No mold | No mold |
| 2 | Lot of mold | Beginning to mold | No mold | No mold | No mold |
| 3 | Dish full of mold | Dish half full of mold | Few colonies of mold beginning | No mold | No mold |
| 4 | Dish full of mold and the mold had turned dark on top. | Dish full of mold | More colonies of mold today than yesterday | One or two colonies of mold beginning | One or two colonies of mold beginning |

These dishes were opened quite a bit for observation. I feel like the propylene glycol in the binder and overspray, as well as the control agar, became contaminated.

The propylene glycol in the binder and overspray is much superior to glycerine and TEO.

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Exhibit D

Sensory Panel Evaluation of 100% BL Cigarettes

I. Cigarettes coded 167-69 (standard glycerine binder BL) versus cigarettes coded 163 (TEG binder BL): A triangle difference smoking test by thirty judges showed no significant differences in taste or preference between the cigarettes.

II. Cigarettes coded 167-69 (standard glycerine binder BL) versus cigarettes coded 164 (propylene glycol binder BL): A triangle difference smoking test by thirty judges showed no significant differences in taste or preference between the cigarettes.

III. Cigarettes coded 164 (propylene binder BL) versus cigarettes coded 169-71 (propylene glycol binder + propylene glycol overspray): A triangle difference smoking test by thirty judges showed the propylene glycol binder BL plus propylene glycol overspray cigarettes definitely milder and preferred.*

(S) C. E. Maxwell, Jr.

* This preference was based on the judges' indication of less irritation from the sample in question.

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Total Particulate Matter Delivery, Mg./Cigt.

| Marl. (Mon #2) | PP Run (A) 163 | PP Run (B) 164 | Control | |
|-------------------|-------------------|-------------------|----------------------|----------------------|
| | | | PP Run (C) 167-69 | PP Run (D) 169-71 |
| 31.1 | 20.0 | 20.0 | 21.7 | 23.2 |
| 28.6 | 21.2 | 22.6 | 20.3 | 22.0 |
| 30.7 | 19.4 | 21.8 | 21.1 | 24.0 |
| 29.9 | 20.5 | 21.5 | 21.5 | 22.3 |
| 26.5 | 22.2 | 18.8 | 19.2 | 19.9 |
| 26.8 | 19.0 | 19.1 | 17.7 | 20.1 |
| 28.5 | 21.2 | 19.5 | 19.7 | 21.9 |
| 27.9 | 20.2 | 18.8 | 19.0 | 21.3 |
| Avg. | 20.5 | 20.3 | 20.0 | 21.8 |
| S.D. | 1.0 | 0.80 | 0.75 | 0.93 |

Nicotine Delivery, Mg./Cigt.

| Marl. (Mon #2) | PP Run (A) 163 | PP Run (B) 164 | Control | |
|-------------------|-------------------|-------------------|----------------------|----------------------|
| | | | PP Run (C) 167-69 | PP Run (D) 169-71 |
| 1.56 | Lost | 0.59 | 0.63 | 0.57 |
| 1.56 | 0.52 | 0.53 | 0.53 | 0.53 |
| 1.55 | 0.52 | 0.58 | 0.61 | 0.57 |
| 1.63 | 0.55 | 0.64 | 0.61 | 0.57 |
| 1.54 | 0.62 | 0.62 | 0.56 | 0.57 |
| 1.54 | 0.54 | 0.58 | 0.55 | 0.47 |
| 1.47 | 0.60 | 0.57 | 0.62 | 0.54 |
| 1.61 | 0.60 | 0.60 | 0.65 | 0.58 |
| Avg. | 0.56 ✓ | 0.59 ✓ | 0.60 ✓ | 0.55 |
| S.D. | 0.06 | 0.04 | 0.05 | 0.04 |

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| | Rejected | Acceptables | Total | |
|--------------------------|----------|-------------|-------|-----|
| Marlboro Monitor | | | 48 | |
| Run 163 (rejects) | 44 | - | 48 | 2.1 |
| Run 163 (acceptables) | 48 | 0.03 | | |
| Run 164 (rejects) | 20 | - | 30 | - |
| Run 164 (acceptables) | 40 | 2.4 | | |
| Run 167-69 (rejects) | 64 | - | 56 | 6.8 |
| Run 167-69 (acceptables) | 48 | 1.3 | | |
| Run 169-71 (rejects) | 56 | - | 54 | 5.9 |
| Run 169-71 (acceptables) | 52 | 0.04 | - | |

| | <u>45.0 gm. Force Reading</u> | | | |
|--------------------------|-------------------------------|------|-----|------|
| Marlboro Monitor | | | 88 | |
| Run 163 (rejects) | 100 | - | 100 | 2.41 |
| Run 163 (acceptables) | 100 | 0 | | |
| Run 164 (rejects) | 92 | - | 96 | - |
| Run 164 (acceptables) | 100 | 2.08 | | |
| Run 167-69 (rejects) | 96 | - | 98 | 0.34 |
| Run 167-69 (acceptables) | 100 | 0.52 | | |
| Run 169-71 (rejects) | 100 | - | 98 | 0.34 |
| Run 169-71 (acceptables) | 96 | 0.52 | | |

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